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ORIGINAL RESEARCH

Hydrolytic and Color Stability of Resin Infiltration: A Preliminary *in vitro* Trial

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ABSTRACT

Aims: Resin infiltration is an emerging technique for management of noncavitated lesions. This study evaluated the *in vitro* hydrolytic and color stability of the ICON® resin infiltration system (IC) in 42 extracted human teeth.

Materials and methods: ICON® resin infiltration system was compared with dental adhesive (DA) and dental sealant (DS). The products were applied according to manufacturer's instructions. The baseline weight and color of the samples were recorded. Color was recorded by spectoral colorimeter. The samples were subjected to four experimental conditions: (1) group 1: Stored in lactic acid solution (pH 4.9) for 24 hours; (2) group 2: Thermocycled for 100 cycles (temperatures: 5°C, 55°C, and dwell time of 15 seconds); (3) group 3: Stored in 0.1 N sodium hydroxide solution (pH 12.48) for 14 days at 60°C; (4) group 4: Stored in phosphate-buffered saline solution (pH 7.2) at 37°C for 4 months. The weight and color were recorded again after removal of the samples from the experimental conditions. Two-factor analysis of variance models and Tukey's Honestly Significant Difference were performed to assess statistical differences among the groups. Scanning electron microscopy imaging was performed for samples from groups 1, 3, and 4.

Results: All the samples showed loss of material and change in color. In the demineralizing solution, IC showed significantly greater weight loss ($p = 0.032$) and color change ($p = 0.038$) compared with DA. Dental Sealant showed significantly greater weight loss than IC ($p = 0.027$) after thermocycling. Teeth in group 3 exhibited the greatest weight loss ($p < 0.001$). Teeth in group 2 exhibited the greatest color change ($p < 0.001$).

Conclusion: All tested materials showed loss of retention and color change in the experimental conditions. Infiltration system exhibited greatest weight loss and color change in demineralizing solution. Dental sealant exhibited greatest weight loss upon thermocycling.

Clinical significance: Clinicians should be cautious about the limitations of retention and color stability when considering resin infiltration for incipient lesions.

Keywords: Color stability, Hydrolytic stability, Resin infiltration, Retention.

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INTRODUCTION

Incipient lesions or white spots are precavitated carious lesions that have an intact surface layer and a porous subsurface layer.[1] They occur on tooth surfaces subjected to demineralizing conditions usually associated with plaque buildup and around orthodontic brackets.[2,3] These lesions represent the initial stage of dental decay and are unaesthetic. Since the surface is intact, conventional tooth preparation involving removal of tooth structure may seem unnecessary or too aggressive. The ICON® resin infiltration system (IC) is a microinvasive procedure that enables penetration of a low-viscosity resin into the porous subsurface layer. This conserves tooth structure because the application of the IC does not require removal of tooth structure with burs. Instead, the IC system requires chemical tooth preparation with hydrochloric acid for 90 seconds, followed by an ethanol-based monomer and finally the application of the resin infiltrant. The ability of resin infiltration to arrest clinical proximal decay has been shown in human studies.[4-6] It has also been reported to mask the unesthetic white spots clinically.[7] In addition to utilizing resin infiltration for precavitated carious lesions, the technique has also been utilized for

Table 1: Application technique of materials used

Technique \ Material	ICON®	Adper single bond plus	Clinpro sealant
Etchant	15% hydrochloric acid for 2 minutes, rinsed for 30 seconds and air-dried	37% phosphoric acid for 15 seconds, rinsed for 10 seconds and dried	37% phosphoric acid for 15 seconds, rinsed for 10 seconds and dried
Procedure Step 1	Icon-Dry for 30 seconds, air-dried	Applied with saturated microbrush, air-thinned for 5 seconds	Applied in single layer with microbrush
Procedure Step 2	Icon infiltrant 3-minute application and 40-second light cure	Light-cured for 20 seconds	Light-cured for 20 seconds
Procedure Step 3	Icon infiltrant 1-minute application and light-cured for 40 seconds		

the esthetic management of teeth affected by fluorosis and hypoplsia[8] as well as developmental defects.[9]

To maintain the low viscosity for infiltration and aesthetic properties, the resin infiltration system remains radioluscent. The clinical studies that have evaluated caries' progression with the resin infiltration system, hence, cannot evaluate the retention of the material by radiographic evaluation.[4-6] While these studies have shown decreased progression of incipient proximal lesions by subtraction radiography, they cannot evaluate any loss or attrition of the resin infiltrant itself by this technique.[4-6] Data on the retention and color stability of the resin infiltration system in *in vitro* conditions to which conventional composite resins are subjected are sparse as well.

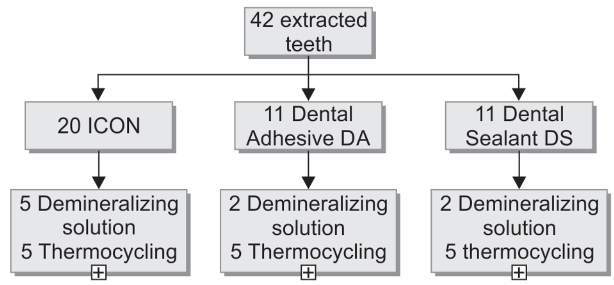
With the purpose of providing better information on the long-term stability of IC from simulated laboratory experiments, the aim of this study was to evaluate the retention and color stability of IC in four experimental conditions.

MATERIALS AND METHODS

Forty-two extracted permanent human teeth were used for this study. The materials tested were ICON® resin infiltration system by DMG America (IC; Englewood, NJ 07631, USA), Adper Single Bond Plus by 3M ESPE (DA; St Paul, MN 55144, USA), and Clinpro Sealant by 3M ESPE (DS; St Paul, MN 55144, USA). The three materials are all relatively low-viscosity resin-based materials that are applied after acid etching of the tooth surface. The materials were applied on the labial surfaces of teeth as per manufacturer's instructions (Table 1). Twenty teeth received IC application, 11 received DA application, and 11 received DS application. The distribution of the samples in the experimental groups is diagrammatically shown in Flow Chart 1. The weights of the teeth were recorded after letting them air-dry at room temperature for 24 hours. The color was recorded with a spectral colorimeter (Chroma Meter CR-400, Konika Minolta Sensing Americas, Ltd, Ramsey NJ 07446, USA). The color was recorded in L*a*b* color space and the change of color (ΔE^*_{ab}) was calculated as per the equation:

$$\Delta E^*_{ab} = \left(\Delta L^*_{ab}{}^2 + \Delta a^*_{ab}{}^2 + \Delta b^*_{ab}{}^2\right)^{1/2}$$

Flow Chart 1: Distribution of samples in experimental groups



After application of the test materials (IC, DA, and DS), the teeth were subjected to four experimental conditions. Group 1 comprised teeth immersed in a demineralizing solution. The demineralizing solution contained lactic acid, calcium phosphate tribasic, and sodium hydroxide and had a pH of 4.9. Five teeth from IC samples, two from DA, and two from DS were immersed in vials containing 5 ml of this demineralizing solution for 24 hours at room temperature. Group 2 comprised teeth that were subjected to 100 cycles of thermocycling. The temperatures were 5°C and 55°C, with a dwell time of 15 seconds. There were five teeth from IC samples, three from DA, and five from DS. Group 3 comprised teeth stored in 5 ml of 0.1 N sodium hydroxide solution (pH 12.48) at 60°C for 14 days. This technique has been shown to simulate resin degradation *in vitro* at a much faster rate than in the clinical oral environment.[10] There were five teeth from IC samples, three from DA, and three from DS. Group 4 comprised teeth immersed in 5 ml phosphate-buffered saline (PBS) solution at 37°C and pH 7.2 for 4 months. There were five teeth from IC samples, three from DA, and three from DS.

After the teeth were taken of out of the experimental conditions, they were allowed to air-dry at room temperature for 24 hours. The weight and color were recorded again. Selected specimens from groups 1, 3, and 4 were also observed under the scanning electron microscope (SEM) for surface topography.

Two-factor analysis of variance (ANOVA) models were used to assess the effect of experimental conditions (demineralization, thermocycling, sodium hydroxide,

Table 2: Weight change (mg) in samples after experimental conditions

Material / *Experiment*	*ICON*	*Adper single bond plus*	*Clinpro sealant*
Demineralization	–3.3 ± 0.20	2.4 ± 0.13	1.2 ± 0.01
Thermocycling	–2.1 ± 0.55	–8.6 ± 0.86	–16.4 ± 0.82
Sodium hydroxide	–56.6 ± 4.36	–67.2 ± 4.7	–36.2 ± 2.95
PBS	2.3 ± 0.34	0.1 ± 0.0	0.6 ± 0.0

Table 3: Color change in samples after experimental conditions

Material / *Experiment*	*ICON*	*Adper single bond plus*	*Clinpro sealant*
Demineralization	10.68 ± 3.7	1.69 ± 0.36	5.85 ± 2.6
Thermocycling	13.54 ± 9.3	20.83 ± 6.27	17.70 ± 3.97
Sodium hydroxide	7.34 ± 6.1	14.12 ± 0.82	6.03 ± 2.25
PBS	6.63 ± 5.4	8.34 ± 9.7	4.89 ± 1.19

and PBS) and material (IC, DA, and DS). *Post hoc* analysis using Tukey's Honestly Significant Difference was performed to assess difference between the four experimental condition groups. The statistical package used was SAS version 9.

RESULTS

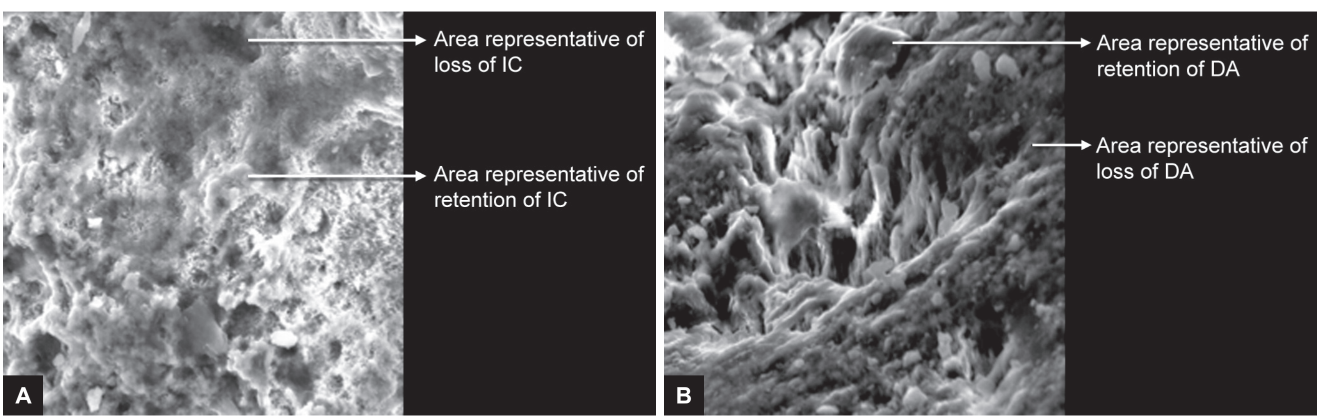
The change in weights of the samples is shown in Table 2. In the demineralizing solution, IC samples showed significantly greater weight loss than DA samples ($p = 0.032$). The difference in weights of the IC and DS samples was not significant ($p = 0.066$). Thermocycling led to weight loss among all the samples, implying material was lost from all the samples. With thermocycling, DS samples showed significantly greater weight loss than IC samples ($p = 0.027$). The difference in weights of the IC and DA samples was not significant ($p = 0.380$). All the samples stored in sodium hydroxide (group 3) exhibited weight loss and there were no differences among the materials tested ($p = 0.761$). All the samples stored in PBS also exhibited weight loss with no difference among the materials tested ($p = 0.597$).

Among the experimental conditions under which the samples were tested, a *post hoc* analysis revealed that group 3 specimens (in sodium hydroxide) exhibited greatest weight loss compared with groups 1, 2, and 4 ($p < 0.001$).

The change in color values (ΔE) is shown in Table 3. The color change for IC samples was significantly higher than that for DA samples stored in the demineralizing solution ($p = 0.038$). There was no significant difference in the color of IC and DS samples stored in the demineralizing solution ($p = 0.257$). All samples in the thermocycling group exhibited color changes and the difference among the three tested materials was not significant ($p = 0.281$). All samples in the sodium hydroxide group exhibited color changes and the difference among the three tested materials was not significant ($p = 0.279$). All samples in the PBS group exhibited color changes and the difference among the three tested materials was not significant ($p = 0.850$).

Among the experimental conditions under which the samples were tested, a *post hoc* analysis revealed that group 2 specimens (thermocycling group) exhibited greatest weight loss compared with groups 1, 3, and 4 ($p < 0.001$).

Samples removed from groups 1, 3, and 4 were studied under SEM for surface quality characteristics. In the SEM images, the smooth white areas are the regions where resin materials were retained and the dark (gray) areas are where enamel was exposed due to loss of the materials. The IC and DA samples from group 1 (Figs 1A and B respectively) showed partial retention of the respective materials, while minimal loss of material was seen in the DS samples in group 1. In group 3, IC samples showed complete loss of material with the etch pattern of the enamel clearly evident (Fig. 2). The DA and DS samples showed partial retention of material. In group 4, IC and DA



Figs 1A and B: (A) Scanning electron microscope view of specimen with IC after exposure to demineralization (2000 × magnification); and (B) SEM view of specimen with DA after exposure to demineralization (2000 × magnification)

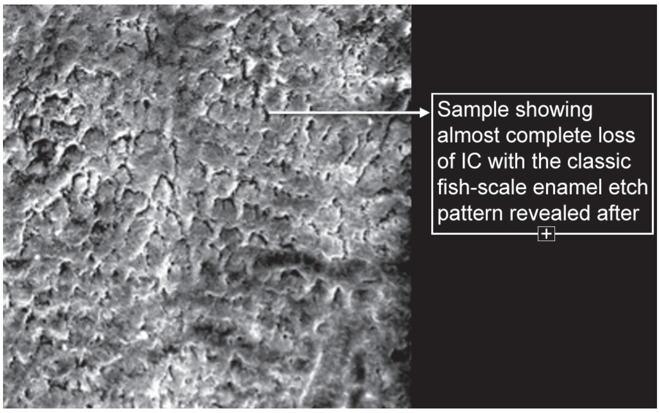
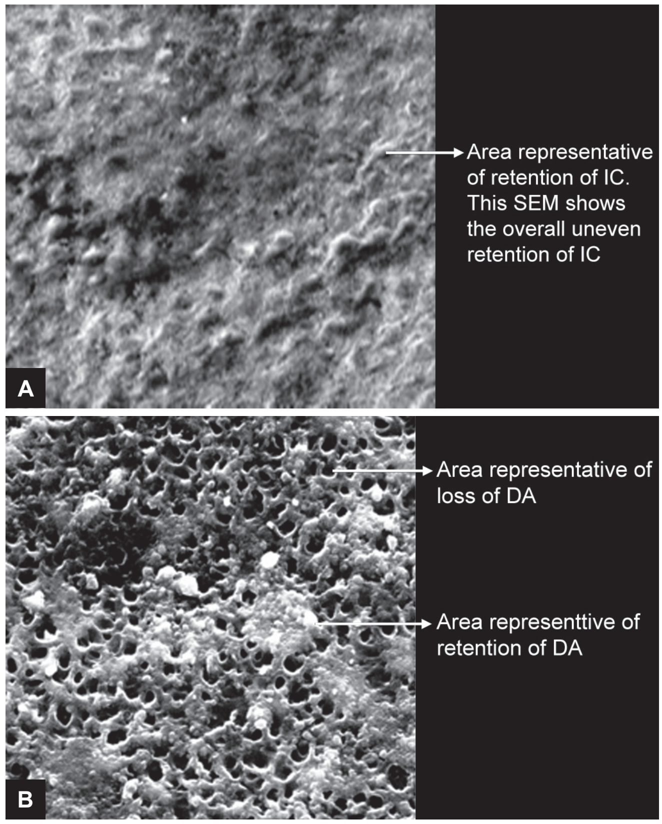


Fig. 2: Scanning electron microscope view of specimen with IC after exposure to sodium hydroxide (1000 × magnification)



Figs 3A and B: (A) Scanning electron microscope view of specimen with IC after exposure to PBS (1000 × magnification); and (B) SEM view of specimen with DA after exposure to PBS (2000× magnification)

samples showed partial loss of material (Figs 3A and B), while DS samples showed minimal loss of material.

DISCUSSION

Evaluating the hydrolytic stability of dental materials is critical in estimating their performance in the dynamic oral cavity. The ICON® resin infiltration system is a relatively new treatment modality for incipient lesions with a few clinical studies showing success of the system in decreasing the progression rate of incipient proximal lesions. However, the retention of IC has not been evaluated under conditions that simulate the oral environment. The need to assess the long-term stability of IC has been identified even in clinical studies, since follow-up duration on clinically treated labial lesions is in the range of weeks to a few months.[9] This *in vitro* study aimed to assess the retention of the material as well as the color stability of IC in four test conditions. We compared IC with commonly used DA and DS, since these materials come closest to IC in viscosity and method of use.

When evaluating for retention of the materials, we used weights of treated samples before and after the experimental conditions to assess the loss of materials. It was found that samples stored in sodium hydroxide showed the greatest loss of material, as this was the harshest of the test conditions. However, even in PBS at body temperature, there was loss of material, indicating that there was degradation of IC, DA, and DS in a fluid medium mimicking physiologic body fluid. The IC-treated samples showed significantly greater material loss in the demineralizing solution. This is of clinical significance because IC is recommended for the treatment of incipient lesions that are caused by demineralization of enamel surface. Hence, the retention of IC can be expected to be compromised in patients who continue to expose their teeth to acidic challenges.

The color stability of test materials was assessed by a spectral colorimeter. With the equipment, we used a ΔE value of 3.7 units or more, which would be perceptible clinically by a dental professional as a color mismatch.[11] All the samples except the DA samples in group 1 showed a ΔE value greater than 3.7. Our study found that IC samples showed significant color change in the demineralizing solution when compared with DA samples. Here, again, it can be inferred that should the acidic challenge persist intraorally, the color match of IC can be expected to deteriorate. There is one report of IC used to mask enamel white spot lesions with satisfactory results for only a 10-month follow-up period.[7] The patient's characteristics regarding continued exposure to acid challenge have not been detailed in this report.[7] When compared with professional fluoride application alone for the management of incipient lesions, IC showed better aesthetic results.[12]

While resin infiltration results in increased enamel microhardness when compared with fluoride exposure alone, it has also been shown that the microhardness of enamel treated with IC is reduced after an acid challenge.[13] This corresponds with the data on material loss seen in our study, confirming that subsequent acid challenge after treatment with IC leads to deterioration of the material. It has also been suggested that an additional surface treatment of the infiltrated resin may enhance the surface properties of IC. It may be a possibility to improve the retention and/or color stability of IC by application of an additional layer of material like glaze and bond.[14] Another method to improve retention under demineralizing conditions may be application of multiple layers of the resin infiltrant or extending the penetration time. It has been shown that extended penetration time of sealants and DAs resulted in reduced lesion progression.[15]

There are limitations to this *in vitro* study. The fluoride exposure and specific history of extracted human teeth used were not known. Variations in teeth characteristics could alter results. The samples' size utilized in our study was small, but still yielded significant results. However, the results need to be verified with a larger sample size. The results of this preliminary *in vitro* study show that more rigorous testing of IC needs to be performed before it is recommended for the routine management of incipient lesions.

CONCLUSION

- The retention and color stability of IC showed significant deterioration in a demineralizing medium.
- Upon thermocycling, DS samples showed the greatest loss of material.
- All the tested materials showed overall loss of retention, with IC samples showing the greatest loss and DS samples showing the least loss.

CLINICAL SIGNIFICANCE

Clinicians should be cautious about the retention and color stability of resin infiltration if they choose to use this technique in the management of incipient carious lesions. Patients receiving this technique should be periodically assessed for retention of the material and color change of the treated surfaces.

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